National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2285

Arson Test Mixture in Methylene Chloride

This Standard Reference Material (SRM) is intended primarily for use in the calibration of chromatographic instrumentation used for the classification of an ignitable liquid residue. This SRM is a solution of 15 compounds, including even carbon number aliphatic hydrocarbons from hexane to tetracosane, toluene, *p*-xylene, 2-ethyltoluene, 3-ethyltoluene, and 1,2,4-trimethylbenzene in methylene chloride. A unit of SRM 2285 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituents: The certified concentrations and estimated uncertainties for the 15 constituents are given in Table 1. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Information Concentration Values: Information values for concentrations, expressed as percent volume fractions, are provided in Table 1 for the 15 components. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value.

Expiration of Certification: The certification of this SRM lot is valid until **31 October 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Preparation and analytical measurements of the SRM were performed by J.V. Goodpaster, B.J. Porter, and M.M. Schantz of the NIST Analytical Chemistry Division and M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

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The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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Analytical Chemistry Division

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Certificate Issue Date: 09 December 2003

John Rumble, Jr, Chief
Measurement Services Division

NOTICE AND WARNING TO USERS

Handling: This material contains alkane and aromatic compounds and should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

INSTRUCTIONS FOR USE

Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified values in Table 1 to be valid within the stated uncertainty. Because of the volatility of methylene chloride, certified values are not applicable to material stored in ampoules that have been opened for more than 2 minutes, even if they are resealed.

PREPARATION AND ANALYSIS[1]

The compounds used in the preparation of this SRM were obtained from Aldrich (Milwaukee, WI), EM Science (Gibbstown, NJ), Mallinkrodt (Phillipsburg, NJ), and JT Baker (Phillipsburg, NJ). The arson test solution was prepared at NIST by weighing and mixing the individual compounds and methylene chloride. The weighed components were added to the methylene chloride and mixed until completely dissolved and homogenized. The total mass of this solution was measured, and the concentrations were calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the purity estimation of each component, which was determined using flame ionization capillary gas chromatography with two stationary phases of different polarities (see Figure 1) and differential scanning calorimetry. This bulk solution was then chilled to approximately -5 °C, and 1.2-mL aliquots were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected using a random stratified sampling scheme, were analyzed in duplicate by using flame ionization capillary gas chromatography with two stationary phases of different polarities. The internal standard, added to each sample for quantification purposes, was *n*-heptadecane. Calibration solutions consisting of weighed amounts of the compounds (adjusted for the purity estimation) and the internal standard compound in methylene chloride were chromatographically analyzed to determine analyte response factors.

The Chemical Abstract Service (CAS) Registry Numbers are given in Table 1, and a representative chromatogram from the flame ionization gas chromatographic analysis on each stationary phase is shown in Figure 1.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Concentrations of Components in SRM 2285

Compound	CAS Registry No.[a]	Certified Concentration (mg/g[b])	Information Concentration (% volume fraction[c])
n-hexane	110-54-3	1.004 ± 0.038	0.199
n-octane	111-65-9	1.130 ± 0.040	0.211
n-decane	124-18-5	1.015 ± 0.023	0.182
n-dodecane	112-40-3	1.371 ± 0.031	0.240
n-tetradecane	629-59-4	1.307 ± 0.030	0.224
n-hexadecane	544-76-3	1.064 ± 0.030	0.180
n-octadecane	593-45-3	1.250 ± 0.043	0.211
n-eicosane	112-95-8	1.382 ± 0.047	0.230
n-docosane	629-97-0	1.356 ± 0.032	0.224
n-tetracosane	646-31-1	1.481 ± 0.046	0.243
toluene	108-88-3	1.249 ± 0.046	0.189
p-xylene	106-42-3	1.360 ± 0.042	0.207
2-ethyltoluene	611-14-3	1.284 ± 0.028	0.191
3-ethyltoluene	620-14-4	1.243 ± 0.026	0.188
1,2,4-trimethylbenzene	95-63-6	1.249 ± 0.031	0.187

[a] Chemical Abstracts, Eleventh Collective Index. Index Guide, American Chemical Society: Columbus, Ohio (1986).

[b] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and chromatographic measurements. The expanded 95 % uncertainty uses a coverage factor of 2 and incorporates both correction for estimated purity and allowance for differences between the concentrations determined by gravimetric preparation and chromatographic measurements [1].

[c] The information concentration value in % volume fraction is provided for user information only. The values were obtained by multiplying the certified values by the measured density of the SRM solution at 22 °C (1.31 g/mL) and dividing by the densities of the individual compounds as reported in the CRC Handbook of Chemistry and Physics, 60th Ed., CRC Press: Boca Raton, Florida (1979).

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed.; ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

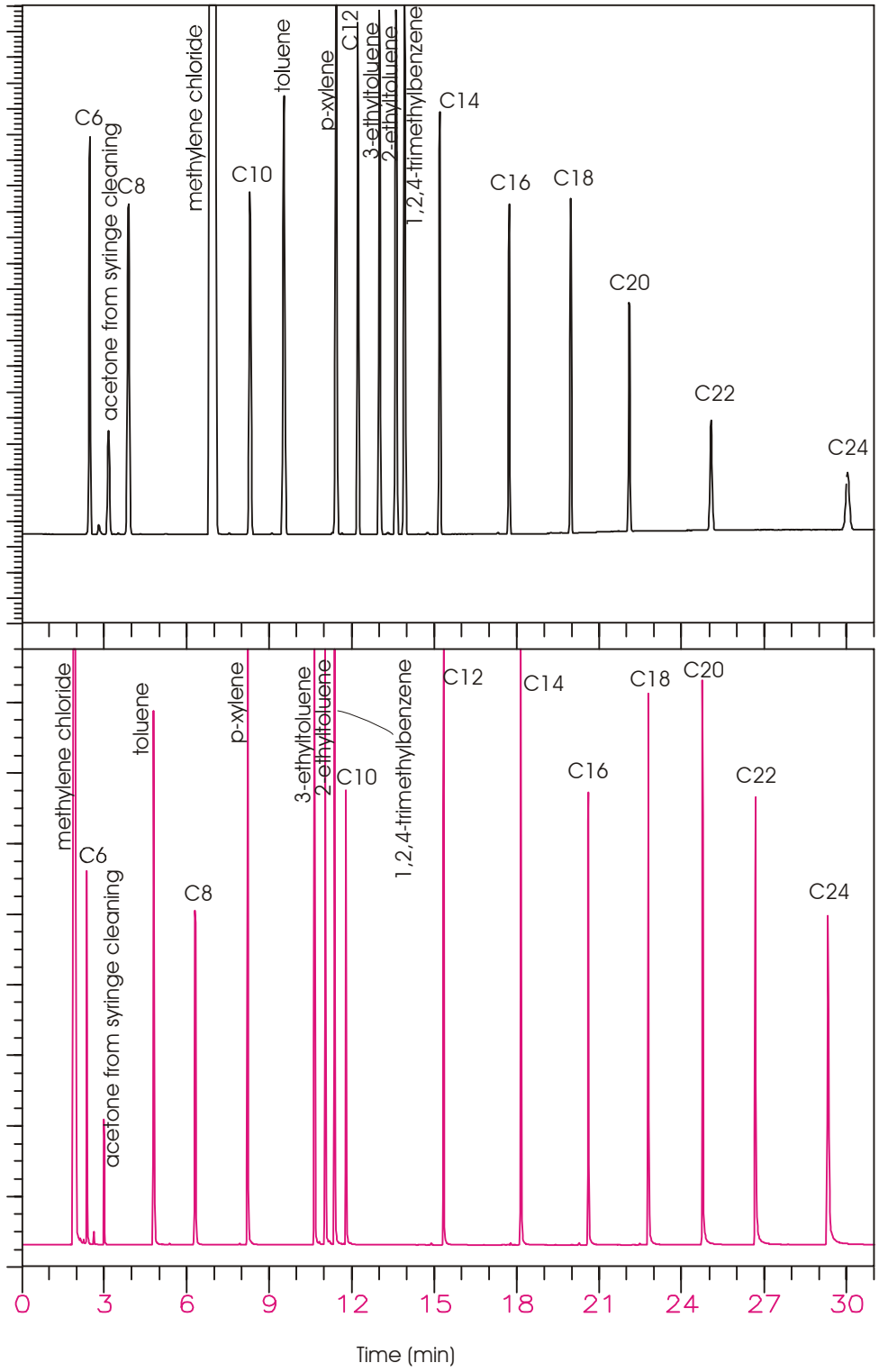

Figure 1. Upper chromatogram: Analysis of SRM 2285 on a polyethylene glycol column (15 m x 0.45 mm id, 0.85 µm film) with a temperature program from 40 °C (5 min hold) to 200 °C (10 min hold) at a rate of 10 °C/min and a constant flow of helium at 1.0 mL/min. Lower chromatogram: Analysis of SRM 2285 on 100 % dimethylpolysiloxane column (30 m x 0.25 mm id, 0.5 µm film) with a temperature program from 55 °C (6 min hold) to 250 °C (7 min hold) at a rate of 10 °C/min and a constant flow of helium at 1.5 mL/min.